INTERNATIONAL STANDARD

ISO 17190-4

First edition
2001-12-01

Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 4: Determination of moisture content by mass loss upon heating

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 4: Détermination de la teneur en humidité au moyen de la perte de masse par chauffage

Reference number
ISO 17190-4:2001(E)

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Printed in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 17190 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 17190-4 was prepared by Technical Committee ISO/TC 173, *Technical systems and aids for disabled or handicapped persons*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

ISO 17190 consists of the following parts, under the general title *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials*:

— *Part 1: Determination of pH*

— *Part 2: Determination of amount of residual monomers*

— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

— *Part 10: Determination of extractable polymer content by potentiometric titration*

— *Part 11: Determination of content of respirable particles*

ISO 17190 is intended to be used in conjunction with ISO 17191, *Urine-absorbing aids for incontinence — Airborne polyacrylate superabsorbent material in the workplace — Determination of the content in respirable dust by sodium atomic absorption spectrometry*.

Annex A of this part of ISO 17190 is given for information only.

Introduction

ISO 17190 consists of a series of test methods originally developed by *European Disposables and Nonwovens Association (EDANA)*. These test methods have been incorporated without technical changes into one International Standard consisting of eleven parts.

These test methods have been in practical use for several years, and have proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). They are applicable to polyacrylate superabsorbent materials, which occur in hygiene products, including urine-absorbing aids for incontinent persons. The test methods are addressed to the *material* exclusively. They are not intended to be used, and are not applicable for use with finished manufactured urine-absorbing aids.

Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 4: Determination of moisture content by mass loss upon heating

1 Scope

This part of ISO 17190 specifies a method for determining the mass loss upon heating for cross-linked polyacrylate (PA) superabsorbent powders with a moisture content ranging between 0 % and 5 % by mass. The method is accurate to within ± 0,1 %.

In general, this method is expected to be applicable to powdered polymeric superabsorbent materials that are free-flowing at temperatures between 15 °C and 150 °C. Substances other than water, that are volatile in this temperature range, will interfere.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 17190. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 17190 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Principle

This procedure determines the mass loss upon dehydration of the test portion in an electrically heated drying oven, kept at (105 ± 2) °C at atmospheric pressure for a period of 3 h.

4 Apparatus

4.1 **Analytical balance**, capable of weighing, to the nearest 0,001 g, masses up to 300 g.

4.2 **Dish**, glass or aluminium, with corresponding removable lid, and with a bottom surface about 50 cm^2.

4.3 **Oven**, thermostatted, capable of maintaining a temperature of (105 ± 2) °C.

4.4 **Desiccator**, with active drying agent (e.g. silica gel).

4.5 **Spatula**, V-shaped, capable of holding about 1 g of PA superabsorbent powder.

5 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take the test sample with a scoop. Place it in an airtight container of adequate size within 3 min after sampling.

Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

6 Procedure

6.1 Place a dish and lid (4.2) into the oven at 105 °C (4.3) for 3 h. While in the oven, remove the lid from the dish. At the end of this heating period, cover the dish with the lid and transfer the lidded dish to the desiccator (4.4). Allow the lidded dish to cool for 30 min to room temperature.

6.2 Weigh, to the nearest 0,001 g, the empty lidded dish and record this mass, in grams, as m_1.

6.3 Remove the lid and, with the help of a V-shaped spatula (4.5), add approximately 4,0 g of a well-mixed representative test portion of PA superabsorbent powder test sample, free from lumps.

6.4 Replace the lid and weigh the lidded dish containing the sample immediately to the nearest 0,001 g. Record this mass, in grams, as m_2.

6.5 Distribute the test portion in a uniform particulate layer over the bottom of the dish, for example by using a spatula and gentle tapping.

6.6 Place the open dish, and its corresponding lid, together in the oven at 105 °C for 3 h.

6.7 After this period, immediately lid the dish before removing it from the oven. Then place it in the desiccator and allow it to cool for 30 min.

6.8 When the lidded dish containing the test portion has cooled to room temperature, remove it from the desiccator and weigh it immediately to the nearest 0,001 g. Record this mass, in grams, as m_3.

6.9 Carry out at least two determinations on the same well-mixed laboratory sample, simultaneously or successively, by the same analyst.

7 Calculation

7.1 Calculate the moisture content w_m, expressed as a percentage, of the test sample using equation (1).

$$w_m = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where

m_1 is the mass, expressed in grams, of the dried empty, lidded dish (6.2);

m_2 is the mass, expressed in grams, of the lidded dish with the test portion before drying (6.4);

m_3 is the mass, expressed in grams, of the lidded dish with test portion after drying (6.8).

7.2 Calculate the average of the two replicate determinations.

8 Precision

The data for the repeatability and reproducibility of this test procedure were established by interlaboratory tests carried-out in 1997 by EDANA and are given in annex A.

The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 0,44 % moisture

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 1,80 % moisture

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If these criteria are still not met, report the results as unusual, then diagnose the source of error for example by verifying correct operation of the instruments and testing a portion of a material with a known value.

9 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

b) the type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

c) a reference to this part of ISO 17190, i.e. ISO 17190-4;

d) the results of moisture content by mass loss for each test portion (7.1), expressed as a mass fraction in percent to the nearest 0,1 %, and the average for duplicate determinations;

e) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 8);

f) any deviations from the procedure or any operations regarded as optional.

Annex A
(informative)

Statistical results of interlaboratory tests

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results as follows:

Sample identification	A	B	C
Number of participating laboratories	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	10	10	10
Number of accepted test results	39	39	40
Mean value (%)	0,62	0,53	3,96
Repeatability standard deviation (s_r)	0,09	0,07	0,16
Repeatability coefficient of variation	15,1 %	12,6 %	4,0 %
Repeatability limit (r) ($2{,}8 \times s_r$)	0,26	0,19	0,44
Reproducibility standard deviation of reproducibility (s_R)	0,13	0,27	0,64
Reproducibility coefficient of variation	20,7 %	51,4 %	16,2 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	0,36	0,77	1,80

ICS 11.180.20

Price based on 4 pages